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■

STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Proposed Standards for Seven Source Categories

ENVIRONMENTAL PROTECTION AGENCY

[40 CFR, Part 60]

STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Proposed Standards for Seven Source Categories

MAY 4, 1973.

Pursuant to section 111 of the Clean Air Act, the Administrator proposes herein standards of performance for new and modified sources within seven categories of stationary sources: Asphalt concrete plants, petroleum refineries, storage vessels for petroleum liquids, secondary lead smelters, secondary brass and bronze ingot production plants, iron and steel plants, and sewage treatment plants. The Administrator also proposes amendments to the general provisions of 40 CFR, part 60, published on December 23, 1971 (36 FR 24876), and to the appendix, "Test Methods," to this part. In a separate publication, on May 2, 1973 (38 FR 10820), the Administrator proposed amendments to the general provisions to prescribe procedures for dealing with emissions which exceed standards during startups, shutdowns, or malfunctions. The general provisions apply to all standards of performance for new and modified sources, both those standards promulgated to date (36 FR 24876) and those to be promulgated in the future.

As prescribed by section 111, this proposal of standards was preceded by the Administrator's determination that these seven categories of sources contribute significantly to air pollution which causes or contributes to the endangerment of public health or welfare and by his publication of a list of these categories of sources in this issue of the FEDERAL REGISTER.

The proposed standards apply to a selected source or sources within each category and to selected air pollutants. For example, the standard pertinent to iron and steel plants applies to the emission of particulate matter from basic oxygen process furnaces.

The bases for the proposed standards include the results of source tests conducted by the Environmental Protection Agency and local agencies, data derived from available technical literature, information gathered during visits to pollution control agencies and plants in the United States and abroad, and comments and suggestions solicited from experts. In each case, the proposed standard reflects the degree-of-emission limitation achievable through the application of the best system of emission reduction which, taking into account the cost of achieving such reduction, the Administrator has determined has been adequately demonstrated. Background information which presents the factors considered in arriving at the proposed standards, including costs and summaries of test data, is available free of charge from the Emission Standards and Engineering Division, Environmental Protection Agency, Research Triangle Park, N.C. 27711, attention: Mr. Don R. Goodwin. It is emphasized that the costs are considered reasonable for new and substantially modified sources and that it is not implied that the same costs apply to the retrofitting of existing sources. Retrofitting existing sources to achieve the proposed emission limitations would in some cases cost much more.

The Environmental Protection Agency has adopted a policy of expressing standards in the metric rather than English system. Although technical terms in test methods 10 and 11 are expressed in metric units, many of those in test methods 1 through 9 are expressed in English units. Test results derived through calculations in test methods 1 through 9 must be converted to metric units to agree with the form of the proposed standards.

Of special interest are the regulations concerning hydrocarbon emissions from storage vessels for petroleum liquids (subpt. K), and the allowable level of particulate emissions from asphalt batch plants (subpt. I).

As explained in technical report 9, emissions of hydrocarbons from storage vessels for petroleum liquids are significant. Most of the hydrocarbon emissions are released during storage and during tank filling. Rates of emissions are dependent on a variety of factors such as the physical properties of the liquid being stored, climatic and meteorological conditions, and the size, type, color, and condition of the tank.

To minimize such losses, normal practice involves the use of floating roof tanks; and when the vapor pressure of the stored hydrocarbon is very high, vapor recovery systems, pressure storage, refrigeration, or combinations thereof. Because of the nature of the emissions (high concentrations for short time periods during tank filling; low concentrations for longer periods during storage), and the configuration of storage tanks, direct emission measurement is highly impracticable, especially for general enforcement purposes. An alternate approach to direct emission measurement is a calculation procedure developed by the American Petroleum Institute to enable the determination of product losses, given such factors as average wind velocity, average ambient diurnal temperature change, product physical characteristics, tank size and mechanical conditions, and volume throughput. This calculation procedure was considered as a possible basis for the standards of performance. Such a procedure, however, if used as the basis for standards of performance, would require plant operators to maintain detailed records on all the parameters used in the calculation, could severely limit flexibility in terms of storage tank usage, and would greatly complicate enforcement procedures. As a practical measure, therefore, the Administrator has determined that equipment specification is the most acceptable approach to standards of performance for storage vessels. The regulations do allow for the use of equivalent technology, provided the same degree of emission control can be demonstrated. The standard, stated in terms of equipment specifications, will achieve essentially the same control as the more complex calculation procedure and will result in a minimum of plant recordkeeping and enforcement problems.

During the development of the proposed performance standard for asphalt concrete plants, considerable comment was received from industry indicating that the allowable emission rate cannot be achieved routinely. Test data, EPA cost analysis for new sources, and other supporting arguments led to the Administrator's judgment that the allowable emission levels can be achieved at a reasonable cost. However, because of the known controversy concerning the proposed standard of performance for asphalt concrete plants, the Administrator urges all interested parties to submit factual data during the comment period to assure that the standards which are promulgated are consistent with the requirements of section 111.

The proposed amendments to subpart A, "General Provisions," include additional abbreviations; a change to the definition of "commenced" which excludes entering into a binding agreement; substitution of an appropriate EPA regional office for the Office of General Enforcement as the addressee for all requests, reports, etc., sent to the Administrator pursuant to this part; and the addition of a provision whereby the Administrator may approve the use of alternative test methods if results show that they are adequate for testing compliance or may waive the requirement for performance tests if it has been demonstrated by other means to his satisfaction that a source is being operated in compliance with the standard.

The purpose of the provision for alternative test methods is to allow, in certain applications, the use of source test methods such as those specified by some State agencies which are sufficiently reliable for certain applications but which may not be, or may not have been shown to be, equivalent to the reference method. For example, an alternative method which does not require traversing during sampling for particulate matter may be approved if such method includes a suitable correction factor designed to account for the error which may result from failing to traverse, or if it can be demonstrated in a specific case that failure to traverse does not affect the accuracy of the test. Similarly, use of an in-stack filter for particulate sampling may be approvable as an alternative method if the method otherwise employs provisions designed to result in precision similar to the compliance method, and a suitable correction factor is included to account for variation between results expected due to filter location. In cases where determination of compliance using an alternative method is disputed, use of the reference method or its equivalent shall be required by the Administrator.

The proposed amendments to the appendix to this part consist of the addition of reference test methods for determining carbon monoxide emissions and hydrogen sulfide concentrations from stationary sources.

In accordance with section 117(f) of the act, publication of these proposed amendments to 40 CFR was preceded by consultation with appropriate advisory committees, independent experts, and Federal departments and agencies. Possible adverse environmental impact resulting from the proposed standards has been considered and determined negligible; a discussion of this subject is included in the background information which will be published at the time of proposal.

Interested persons may participate in this rulemaking by submitting written comments (in triplicate) to the Emission Standards and Engineering Division, Environmental Protection Agency, Research Triangle Park, N.C. 27711, attention: Mr. Don R. Goodwin. The Administrator will welcome comments on all aspects of the proposed regulations, including economic and technological issues, and on the proposed test methods. All relevant comments received not later than July 26, 1973, will be considered. Receipt of comments will be acknowledged, but the Emission Standards and Engineering Division will not provide substantive response to individual comments. The standards, modified if and as the Administrator deems appropriate after consideration of comments, will be promulgated no later than September 10, 1973, as required by the act. Comments received will be available for public inspection at the Office of Public Affairs, 401 M Street SW., Washington, D.C. 20460.

This notice of proposed rulemaking is issued under the authority of sections 111 and 114 of the Clean Air Act, as amended (42 U.S.C. 1857c–6 and 9).

Dated June 1, 1973.

ROBERT W. FRI,
Acting Administrator,
Environmental Protection Agency.

It is proposed to amend part 60 of chapter I, title 40, of the Code of Federal Regulations as follows:

1. Section 60.2 is amended by deleting the words "binding agreement or" from paragraph (i) and by adding paragraphs (p), (q), (r) and (s). As amended, § 60.2 reads as follows:

§ 60.2 Definitions.

* * * * *

(i) "Commenced" means that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

* * * * *

(p) "Reference method" means a method of sampling and analyzing for an air pollutant, as described in the appendix to this part.

(q) "Equivalent method" means any method of sampling and analyzing for an air pollutant which is demonstrated to the Administrator's satisfaction to have a consistent and quantitative relationship to the reference method under specified conditions.

(r) "Alternative method" means a method which does not meet all the criteria for equivalency by which has been demonstrated to the Administrator's satisfaction to, in specific cases, produce results adequate for his determination of compliance.

(s) "Nm^3" means dry cubic meters at normal conditions.

2. In § 60.3, new abbreviations are added as follows:

§ 60.3 Abbreviations.

* * * * *

sec—second.
ppm—parts per million.
H_2O—water.
CO—carbon monoxide.
mv—millivolt.
N_2—nitrogen.
C or °C—degree centigrade.
F or °F—degree Fahrenheit.
R or °R—degree Rankine.
K or °K—degree Kelvin.
ppb—parts per billion.
HCl—hydrochloric acid.
CdS—cadmium sulfide.
mol. wt—molecular weight.
dscf—dry standard cubic feet.
eq—equivalents.
meq—milliequivalents.
g eq—gram equivalents.
O_2—oxygen.
H_2S—hydrogen sulfide.
m—meter.
m^2—square meter.
m^3—cubic meter.
N—standard or normal conditions.

3. Section 60.4 is revised to read as follows:

§ 60.4 Address.

All requests, reports, applications, submittals, and other communications to the Administrator pursuant to this part shall be submitted in duplicate and addressed to the appropriate Regional Office of the Environmental Protection Agency, to the attention of the Director, Enforcement Division. The regional offices are as follows:

Region I (Connecticut, Maine, New Hampshire, Massachusetts, Rhode Island, Vermont), John F. Kennedy Federal Bldg., Boston, Mass. 02203.
Region II (New York, New Jersey, Puerto Rico, Virgin Islands), Federal Office Bldg., 26 Federal Plaza (Foley Square), New York, N.Y. 10007.
Region III (Delaware, District of Columbia, Pennsylvania, Maryland, Virginia, West Virginia), Curtis Bldg., Sixth and Walnut Sts., Philadelphia, Pa. 19106.
Region IV (Alabama, Florida, Georgia, Mississippi, Kentucky, North Carolina, South Carolina, Tennessee), suite 300, 1421 Peachtree St., Atlanta, Ga. 30309.
Region V (Illinois, Indiana, Minnesota, Michigan, Ohio, Wisconsin), 1 North Wacker Dr., Chicago, Ill. 60606.
Region VI (Arkansas, Louisiana, New Mexico, Oklahoma, Texas), 1600 Patterson St., Dallas, Tex. 75201.
Region VII (Iowa, Kansas, Missouri, Nebraska), 1735 Baltimore St., Kansas City, Mo. 64108.
Region VIII (Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming), 916 Lincoln Towers, 1860 Lincoln St., Denver, Colo. 80203.
Region IX (Arizona, California, Hawaii, Nevada, Guam, American Samoa), 100 California St., San Francisco, Calif. 94111.
Region X (Washington, Oregon, Idaho, Alaska), 1200 Sixth Ave., Seattle, Wash. 98101.

4. In § 60.8, paragraph (b) is revised to read as follows:

§ 60.8 Performance tests.

* * * * *

(b) Performance tests shall be conducted and data reduced in accordance with the procedures contained in the applicable reference test method appended to this part unless the Administrator (1) approves the use of an equivalent method, (2) approves the use of an alternative method the results of which he has determined to be adequate for indicating whether a specific source is in compliance, or (3) waives the requirement for performance tests because the owner or operator of a source has demonstrated by other means to the Administrator's satisfaction that the affected facility is being operated in compliance with the standard. Nothing in this subparagraph shall be construed to abrogate the Administrator's authority to require testing under section 114 of the act.

* * * * *

5. Subparts I, J, K, L, M, N, and O are added, as follows:

Subpart I—Standards of Performance for Asphalt Concrete Plants

Sec.
60.90 Applicability and designation of affected facility.
60.91 Definitions.
60.92 Standard for particulate matter.
60.93 Emission records.
60.94 Test methods and procedures.

AUTHORITY.—Secs. 111 and 114 of the Clean Air Act, as amended (42 U.S.C. 1857c–6 and 9).

Subpart I—Standards of Performance for Asphalt Concrete Plants

§ 60.90 Applicability and designation of affected facility.

The provisions of this subpart are applicable to the following affected facilities in asphalt concrete plants: Dryers, hot aggregate elevators, screening (classifying systems, hot aggregate storage systems, hot aggregate weighing systems, asphalt concrete mixing systems, mineral filler loading systems, transfer and storage systems, and the loading, transfer, and storage systems which are associated with emission control systems.

§ 60.91 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Asphalt concrete plant" means any facility manufacturing asphalt concrete by heating and drying aggregate and mixing with asphalt cements.

(b) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by method 5.

§ 60.92 Standard for particulate matter.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated, but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge of gases into the atmosphere from any affected facility which:

(a) Contain particulate matter in excess of 70 mg/N m³ (0.031 gr/dscf).

(b) Exhibit 10 percent opacity, or greater, except for 2 minutes in any one hour. Where the presence of uncombined water is the only reason for failure to meet the requirements of this subparagraph, such failures shall not be a violation of this section.

§ 60.93 Emission records.

The owner or operator of any affected facility subject to the provisions of the subpart shall maintain a file of any particulate matter emission measurements. The record(s) shall be retained for at least 2 years following the dates on which the tests were conducted.

§ 60.94 Test methods and procedures.

(a) The provisions of this section apply to performance tests for determining compliance with the standard prescribed by § 60.92.

(b) All performance tests shall be conducted while the affected facility being tested is operating at or above the maximum production rate at which such facility will be operated and/or under such other conditions as the Administrator shall specify in order to achieve valid test results.

(c) Compliance with the standard shall be determined by sampling and observing undiluted gases. If air or other gaseous diluent is added prior to a sampling or observation point, the owner or operator shall determine the amount of dilution by a means acceptable to the Administrator.

(d) The reference methods for conducting performance tests are appended to this part.

(1) Method 5 shall be used for determining concentration of particulate matter and moisture, method 1 for traversing, method 2 for determining the volumetric flow rate, and method 3 for gas analysis. The sampling time shall be not less than 60 minutes and not more than 150 minutes, and the minimum sampling rate shall be 0.9 dry standard cubic meter per hour (0.53 dscfm).

Subpart J—Standards of Performance for Petroleum Refineries

Sec.

60.100 Applicability and designation of affected facility.
60.101 Definitions.
60.102 Standard for particulate matter.
60.103 Standard for carbon monoxide.
60.104 Standard for sulfur dioxide.
60.105 Emission monitoring.
60.106 Test methods and procedures.

AUTHORITY.—Secs. 111 and 114 of the Clean Air Act, as amended (42 U.S.C. 1857c–6 and 9).

Subpart J—Standards of Performance for Petroleum Refineries

§ 60.100 Applicability and designation of affected facility.

The provisions of this subpart are applicable to the following affected facilities in petroleum refineries: Fluid catalytic cracking unit catalyst regenerators, process heaters, boilers, and waste gas disposal systems.

§ 60.101 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Petroleum refinery" means any facility in which crude petroleum is refined, processed, or otherwise undergoes a chemical or physical change.

(b) "Crude petroleum" means a mixture consisting of hydrocarbons and/or sulfur, nitrogen and/or oxygen derivatives of hydrocarbons, which is usually naturally occurring and removed from the earth in the liquid state.

(c) "Hydrocarbon" means any material containing carbon and hydrogen.

(d) "Process gas" means a gaseous mixture of hydrocarbons produced by a refinery process unit.

(e) "Fuel gas" means process gas and/or natural gas or any other gaseous mixture which will support combustion, but does not include stack gases from fluid catalytic cracking unit catalyst regenerators.

(f) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by method 5.

(g) "Refinery process unit" means any segment of the petroleum refinery in which a specific processing operation is conducted.

(h) "Waste gas disposal system" means any grouping of equipment or contrivances used to burn or otherwise vent process gas to the atmosphere but does not include facilities where conversion to sulfur or sulfuric acid is utilized primarily as a means of preventing emissions to the atmosphere of sulfur compounds and does not include equipment subject to subpart H of this part.

§ 60.102 Standard for particulate matter.

(a) On or after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere from the fluid catalytic cracking unit catalyst regenerator any gases which:

(1) Contain particulate matter in excess of 50 mg/N m³ (0.022 gr/dscf).

(2) Exhibit 20 percent opacity or greater, except for 3 minutes in any one hour. Where the presence of uncombined water is the only reason for failure to meet the requirements of this subparagraph, such failure shall not be a violation of this section.

(b) In those instances where auxiliary liquid or solid fuels are burned in an incinerator-waste heat boiler, particulate matter in excess of that allowed by paragraph (a)(1) of this section may be emitted to the atmosphere except that the incremental rate of particulate emissions shall not exceed 0.18 g/million calories (0.10 lb per million Btu) of heat input attributable to such liquid or solid fuel.

§ 60.103 Standard for carbon monoxide.

On or after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere from the fluid catalytic cracking unit catalyst regenerator any gases which contain CO in excess of 0.050 percent by volume.

§ 60.104 Standard for sulfur dioxide.

(a) On or after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall burn, in any affected facility subject to the provisions of this subpart, or release to the atmosphere any process gas which contains H_2S in excess of 230 mg/N m³ (0.10 gr/dscf) of fuel gas, except as provided in paragraph (b) of this section.

(b) The owner or operator may elect to treat gases resulting from the combustion of any process gas in a manner which prevents the release of SO_2 to the atmosphere as effectively as compliance with the requirements of paragraph (a) of this section.

§ 60.105 Emission monitoring.

(a) The owner or operator of any petroleum refinery subject to the provisions of this subpart shall install, calibrate, maintain, and operate gas concentration or other monitoring instruments as applicable:

(1) A photoelectric or other type smoke detector and recorder to continuously monitor the opacity of particulate matter released to the atmosphere.

(2) An instrument for continuously monitoring and recording the concentration of CO in gases released to the atmosphere from fluid catalytic cracking unit catalyst regenerators except where compliance is achieved through the combustion of CO and O_2 concentration and temperature are monitored in accordance with paragraph (a)(3) of this section.

(3) Instruments for continuously monitoring and recording firebox temperature and O_2 content of the exhaust gas from any CO combustion device except where the requirements for paragraph (a)(2) of this section are met.

(4) An instrument for continuously monitoring and recording concentrations of H_2S in process gases burned in any

affected facility except where the requirements of § 60.104(b) are met.

(5) An instrument for continuously monitoring and recording concentrations of SO_2 in process gas combustion gases from any affected facility except where the requirements of § 60.104(a) are met.

(b) Instruments and sampling systems installed and used pursuant to this section shall meet specifications prescribed by the Administrator and each instrument shall be calibrated in accordance with the method prescribed by the manufacturer of such instrument. The instruments shall be subjected to the manufacturers' recommended zero adjustment and calibration procedures at least once per 24-hour-operating period unless the manufacturer specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed.

(c) Production rate and hours of operation for any fluid catalytic cracking unit catalyst regenerator shall be recorded daily.

(d) The owner or operator of any petroleum refinery subject to the provisions of this part shall maintain a file of all measurements required by this part and any particulate matter emission measurements. Appropriate measurements shall be reduced to the units of the applicable standard daily and summarized monthly. The record of any such measurements and summary shall be retained for at least 2 years following the date of such measurements and summaries.

§ 60.106 Test methods and procedures.

(a) The provisions of this section apply to performance tests for determining compliance with the standards prescribed by §§ 60.102, 60.103, and 60.104.

(b) All performance tests shall be conducted while the affected facility being tested is operating at or above the maximum production rate at which such facility will be operated and/or under such other conditions as the Administrator shall specify in order to achieve valid test results.

(c) Compliance with the standard shall be determined by sampling and observing undiluted gases. If air or other gaseous diluent is added prior to a sampling or observation, the owner or operator shall determine the amount of dilution by a means acceptable to the Administrator.

(d) The reference methods for conducting performance tests are appended to this part.

(1) Method 5 shall be used for determining concentration of particulate matter and moisture. The sampling time shall be not less than 60 minutes and not more than 150 minutes, and the minimum sampling rate shall be 0.9 dry standard cubic meter per minute (0.53 dscfm).

(2) Method 10 shall be used for determining concentration of CO. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The sampling time shall be not less than 60 minutes and not more than 150 minutes.

(3) Method 6 shall be used for determining concentration of SO_2, except that H_2S concentration of the fuel gas may be determined instead. Method 4 shall be used to determine moisture content. The sampling site shall be the same as for determining volumetric flow rate. The sampling point in the duct shall be at the centroid of the cross section if the cross-sectional area is less than 5 m^2 (54 ft^2) or at a point no closer to the walls than 1 m (29 inches) if the cross sectional area is 5 m^2 or more. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The sampling time shall be no less than 20 minutes and no more than 60 minutes, and minimum sampling volume shall be 0.02m^3 (0.71 ft^3) corrected to standard conditions. Two samples shall constitute one repetition and shall be taken at 1-hour intervals.

(4) Method 11 shall be used for determining the concentration of H_2S in fuel gas. The sampling site and point shall be located at the centroid of the fuel gasline. For refinery fuel gaslines operating at pressures substantially above atmospheric pressure, the sample must be reduced to nominally atmospheric pressure before attempting to introduce the sample into the train. This may be done with a flow control valve. If the pressure is high enough to operate the train without a vacuum pump, the pump may be eliminated from the train. The sampling rate shall not exceed 0.084 N m^3h (3 scfh). Four samples shall be taken at intervals of at least 30 minutes for a sampling time of not less than 60 minutes and not more than 150 minutes.

(5) Traversing shall be conducted according to method 1, and method 2 shall be used to determine volumetric flow rate of the total effluent. Method 3 shall be used for gas analysis whenever tests using method 5, 6, or 10 are conducted.

Subpart K—Standards of Performance for Storage Vessels for Petroleum Liquids

Sec.

60.110 Applicability and designation of affected facility.
60.111 Definitions.
60.112 Standard for hydrocarbons.
60.113 Monitoring of operations.
60.114 Storage vessel maintenance.

AUTHORITY.—Secs. 111 and 114 of the Clean Air Act, as amended (42 U.S.C. 1857c-6 and 9).

Subpart K—Standards of Performance for Storage Vessels for Petroleum Liquids

§ 60.110 Applicability and designation of affected facility.

The provisions of this subpart are applicable to each storage vessel for petroleum liquids of more than 245,000 l (65,000 gal) capacity, which is the affected facility.

§ 60.111 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Storage vessel" means any tank, reservoir or container used for the storage of petroleum liquids, but does not include underground tanks.

(b) "Petroleum liquids" means crude petroleum or any derivative thereof.

(c) Crude petroleum" means a mixture consisting of hydrocarbons and/or sulfur, nitrogen and/or oxygen derivatives of hydrocarbons, which is usually naturally occurring and removed from the Earth in the liquid state.

(d) "Petroleum distillate" means finished and intermediate products which are manufactured in crude petroleum processing and refining operations.

(e) "True vapor pressure" means the equilibrium pressure exerted by a hydrocarbon at any given temperature.

(f) "Hydrocarbon" means any material containing carbon and hydrogen.

(g) "Floating roof" means a double deck or flexible single deck pontoon type storage vessel cover, which rests upon and is supported by the petroleum liquid being contained.

(h) "Vapor recovery system" means a vapor gathering system capable of collecting hydrocarbon vapors and gases discharged and a vapor disposal system capable of processing such hydrocarbon vapors and gases so as to prevent their emission to the atmosphere.

(i) "Conservation vent" means a breather valve or pressure-vacuum relief valve used as an accessory for a vent opening.

§ 60.112 Standard for hydrocarbons.

No owner or operator subject to the provisions of this part shall place, hold, or store in a storage vessel any petroleum liquid which has a true vapor pressure (under actual storage conditions) during such storage which is:

(a) 78 mm Hg (1.52 lb/in^2a) or less unless the storage vessel is equipped with a conservation vent or its equivalent.

(b) In excess of 78 mm Hg (1.52 lb/in^2a) but not greater than 570 mm Hg (11.1 lb/in^2a) unless the storage vessel is equipped with a floating roof or its equivalent.

(c) In excess of 570 mm Hg (11.1 lb/in^2a) unless the storage vessel is equipped with a vapor recovery system or its equivalent.

§ 60.113 Monitoring of operations.

(a) The owner or operator of any storage vessel subject to the provisions of this part shall maintain a file of daily petroleum liquid transfer, bulk petroleum liquid temperature, and petroleum liquid true vapor pressure at the bulk liquid temperature. The type of petroleum liquid, quantity transferred, bulk temperature, and true vapor pressure shall be summarized monthly. The record(s) and summary shall be retained for at least 2 years following the date of such records and summaries. This requirement shall not apply to:

(1) Petroleum liquids which have a true vapor pressure at actual storage conditions of 26 mm Hg (0.5 lb/in^2a) or less or

(2) Petroleum liquids which have a true vapor pressure at actual storage conditions between 100 and 470 mm Hg inclusively (1.94 and 9.1 lb/in²a).

(b) The true vapor pressure at the bulk liquid temperature shall be determined in accordance with American Petroleum Institute Bulletin 2517, Evaporation Loss from Floating Roof Tanks.

§ 60.114 Storage vessel maintenance.

No owner or operator subject to the provisions of this part shall place, hold, or store in a storage vessel any petroleum liquid which has a true vapor pressure at actual storage conditions which is in excess of 78 mm Hg (1.52 lb/in²a) unless:

(a) It is painted and maintained so as to prevent excessive temperature and vapor pressure increases,

(b) The seals on any floating roof are maintained so as to minimize emissions, and

(c) All gaging and sampling devices are gas-tight except when gaging or sampling is taking place.

Subpart L—Standards of Performance for Secondary Lead Smelters

Sec.

60.120 Applicability and designation of affected facility.
60.121 Definitions.
60.122 Standard for particulate matter.
60.123 Emission records.
60.124 Test methods and procedures.

AUTHORITY.—Secs. 111 and 114 of the Clean Air Act, as amended (42 U.S.C. 1857c-6 and 9).

Subpart L—Standards of Performance for Secondary Lead Smelters

§ 60.120 Applicability and designation of affected facility.

The provisions of this subpart are applicable to the following affected facilities in secondary lead smelters: blast (cupola) furnaces, reverberatory furnaces, and pot furnaces of more than 250 kg (550 lb) charging capacity.

§ 60.121 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Reverberatory furnace" means any stationary, rotating, rocking, or tilting type reverberatory furnaces.

(b) "Secondary lead smelter" means any facility producing lead from a lead-bearing scrap material by smelting to the metallic form.

(c) "Lead" means elemental lead or alloys in which the predominating component is lead.

(d) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by method 5.

§ 60.122 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere from a blast (cupola) or reverberatory furnace any gases which:

(1) Contain particulate matter in excess of 50 mg/Nm^3 (0.022 gr/dscf).

(2) Exhibit 20 percent opacity or greater except for 2 minutes in any one hour.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere from any pot furnace any gases which exhibit 10 percent opacity or greater except for 2 minutes in any one hour.

(c) Where the presence of uncombined water is the only reason for failure to meet the requirements of paragraph (a) (2) or (b) of this section, such failure shall not be a violation of this section.

§ 60.123 Emission records.

The owner or operator of any furnace subject to the provisions of this subpart shall maintain a file of all measurements required by this subpart. The record of any such measurements and summary shall be retained for at least 2 years following the date of such measurements and summaries.

§ 60.124 Test methods and procedures.

(a) The provisions of this section apply to performance tests for determining compliance with the standard prescribed by § 60.162.

(b) All performance tests shall be conducted while the affected facility being tested is operating at or above the maximum production rate at which such facility will be operated and/or under such other conditions as the Administrator shall specify in order to achieve valid test results.

(c) Compliance with the standard shall be determined by sampling or observing undiluted gases. If air or other gaseous diluent is added prior to a sampling or observation point, the owner or operator shall determine the amount of dilution by a means acceptable to the Administrator.

(d) The reference methods for conducting performance tests are appended to this part.

(1) Method 5 shall be used for determining concentration of particulate matter and moisture, method 1 for traversing, method 2 for determining the volumetric flow rate, and method 3 for analysis. The sampling time shall be not less than 60 minutes and not more than 150 minutes, and the minimum sampling rate shall be 0.9 dry standard cubic meter per hour (0.53 dscfm).

Subpart M—Standards of Performance for Secondary Brass and Bronze Ingot Production Plants

Sec.

60.130 Applicability and designation of affected facility.
60.131 Definitions.
60.132 Standard for particulate matter.
60.133 Emission records.
60.134 Test methods and procedures.

AUTHORITY.—Secs. 111 and 114 of the Clean Air Act, as amended (42 U.S.C. 1857c-6 and 9).

Subpart M—Standards of Performance for Secondary Brass and Bronze Ingot Production Plants

§ 60.130 Applicability and designation of affected facility.

The provisions of this subpart are applicable to the following affected facilities in secondary brass or bronze ingot production plants: Reverberatory and electric furnaces of 1,000 kg (2,205 lb) or greater production capacity and blast (cupola) furnaces of 250 kg/h (550 lb/h) or greater production capacity.

§ 60.131 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Brass and bronze" means any metal alloy containing copper as its predominant constituent, and lesser amounts of zinc, tin, lead, or other metals.

(b) "Reverberatory furnace" means any stationary, rotating, rocking or tilting type reverberatory furnace.

(c) "Electric furnace" means any furnace which uses electricity to produce over 50 percent of the heat required in the production of refined brass or bronze.

(d) "Blast furnace" means any furnace used to recover metal from slag.

(e) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by method 5.

§ 60.132 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere from a reverberatory furnace any gases which:

(1) Contain particulate matter in excess of 50 mg/N m^3 (0.022 gr/dscf).

(2) Exhibit 10 percent opacity or greater except for 2 minutes in any one hour.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere from a blast (cupola) or electric furnace any gases which exhibit 10 percent opacity or greater except for 2 minutes in any one hour.

(c) Where the presence of uncombined water is the only reason for failure to meet the requirements of paragraphs (a) (2) or (b) of this section, such failure shall not be a violation of this section.

§ 60.133 Emission records.

The owner or operator of any affected facility subject to the provisions of this

part shall maintain a file of any particulate matter emission measurements. The record(s) shall be retained for at least 2 years following the dates on which the tests were conducted.

§ 60.134 Test methods and procedures.

(a) The provisions of this section apply to performance tests for determining compliance with the standard prescribed by § 60.132.

(b) All performance tests shall be conducted while the affected facility being tested is operating at or above the maximum production rate at which such facility will be operated and/or under such other conditions as the Administrator shall specify in order to achieve valid test results.

(c) Compliance with the standard shall be determined by sampling and observing undiluted gases. If air or other gaseous diluent is added prior to a sampling or observation, the owner or operator shall determine the amount of dilution by a means acceptable to the Administrator.

(d) The reference methods for conducting performance tests are appended to this part.

(1) Method 5 shall be used for determining concentration of particulate matter and moisture, method 1 for traversing, method 2 for determining the volumetric flow rate, and method 3 for gas analysis. The sampling time shall be not less than 60 minutes and not more than 150 minutes, and the minimum sampling rate shall be 0.9 dry standard cubic meter per hour (0.53 dscfm).

Subpart N—Standards of Performance for Iron and Steel Plants

Sec.
60.140 Applicability and designation of affected facility.
60.141 Definitions.
60.142 Standard for particulate matter.
60.143 Emission monitoring.
60.144 Test methods and procedures.

AUTHORITY.—Secs. 111 and 114 of the Clean Air Act, as amended (42 U.S.C. 1857c–6 and 9).

Subpart N—Standards of Performance for Iron and Steel Plants

§ 60.140 Applicability and designation of affected facility.

The provisions of this subpart are applicable to each basic oxygen process furnace, which is the affected facility.

§ 60.141 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Basic oxygen process furnace" (BOPF) means any furnace producing steel by charging scrap steel, hot metal, and flux materials into a vessel and introducing a high volume of an oxygen-rich gas.

(b) "Heat" means the quantity of steel produced in one batch.

(c) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by method 5.

§ 60.142 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180 days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere from any affected facility any gases which:

(1) Contain particulate matter in excess of 50 mg/Nm^3 (0.022 gr/dscf).

(2) Exhibit 10 percent opacity or greater, except for 2 minutes in any one hour. Where the presence of uncombined water is the only reason for failure to meet the requirements of this subparagraph, such failure shall not be a violation of this section.

§ 60.143 Emission monitoring.

(a) The owner or operator of any affected facility subject to the provisions of this subpart shall install, calibrate, maintain, and operate a photoelectric or other type smoke detector and recorder to continuously monitor the opacity of particulate matter released to the atmosphere and shall retain the records for at least 2 years from the dates on which the data were recorded.

(b) The instrument installed and used pursuant to this section shall meet specifications prescribed by the Administrator and shall be calibrated in accordance with the method(s) prescribed by the manufacturer of such instrument. The instrument shall be subjected to the manufacturer's recommended zero adjustment and calibration procedures at least once per 24-hour operating period unless the manufacturer specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed.

(c) The owner or operator of any BOPF subject to the provisions of this part shall maintain a file of any particulate matter emission measurements. The record(s) shall be retained for at least 2 years from the dates on which the tests were conducted.

§ 60.144 Test methods and procedures.

(a) The provisions of this section apply to performance tests for determining compliance with the standard prescribed by § 60.142.

(b) All performance tests shall be conducted while the affected facility being tested is operating at or above the maximum production rate at which such facility will be operated and/or under such other conditions as the Administrator shall specify in order to achieve valid test results.

(c) Compliance with the standard shall be determined by sampling and observing undiluted gases. If air or other gaseous diluent is added prior to a sampling or observation point, the owner or operator shall determine the amount of dilution by a means acceptable to the Administrator.

(d) The reference methods for conducting performance tests are appended to this part.

(1) Method 5 shall be used for determining concentration of particulate matter and moisture, method 1 for traversing, method 2 for determining the volumetric flow rate, and method 3 for gas analysis. The minimum total sampling time shall be four heats, and the minimum sampling rate shall be 0.9 dry standard cubic meter per hour (0.53 dscfm). Sampling shall start at the beginning of each scrap preheat or oxygen blow and shall terminate immediately prior to tapping.

Subpart O—Standards of Performance for Sewage Treatment Plants

Sec.
60.150 Applicability and designation of affected facility.
60.151 Definitions.
60.152 Standard for particulate matter.
60.153 Emission records.
60.154 Test methods and procedures.

AUTHORITY.—Secs. 111 and 114 of the Clean Act, as amended (42 U.S.C. 1857c–6 and 9).

Subpart O—Standards of Performance for Sewage Treatment Plants

§ 60.150 Applicability and designation of affected facility.

The provisions of this subpart are applicable to each sewage sludge incinerator, which is the affected facility.

§ 60.151 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Sewage sludge incinerator" means any combusition device used in the process of burning sewage sludge for the primary purpose of solids sterilization and to reduce the volume of waste by removing combustible matter, but does not include portable facilities or facilities used solely for burning scum or other floatable materials, recalcining lime, or regenerating activated carbon.

(b) "Sewage sludge" means the solid waste byproduct of municipal sewage treatment processes, including any solids removed in any unit operation of such treatment process.

(c) "Sewage treatment plant" means any arrangement of devices and structures for the treatment of sewage and all appurtenances used for treatment and disposal of sewage and other waste byproducts.

(d) "Sewage" means the spent water of a community consisting of a combination of liquid- and water-carried wastes from residences, commercial buildings, industrial plants, and institutions, together with any ground water, surface water, and storm water that may be present.

(e) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by method 5.

§ 60.152 Standard for particulate matter.

On or after the date on which the performance test required to be conducted by § 60.8 is initiated but no later than 180

days after initial startup, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere any gases which:

(a) Contain particulate matter in excess of 70 mg/Nm³ (0.031 gr/dscf).

(b) Exhibit 10 percent opacity or greater, except for 2 minutes in any one hour. Where the persence of uncombined water is the only reason for failure to meet the requirements of this subparagraph, such failure shall not be a violation of this section.

§ 60.153 Emission records.

The owner or operator shall maintain a file of all measurements required by this subpart. The record of any such measurement and summary shall be retained at least 2 years following the date of such measurements and summaries.

§ 60.154 Test methods and procedures.

(a) The provisions of this section apply to performance tests for determining compliance with the standard prescribed by § 60.152.

(b) All performance tests shall be conducted while the affected facility being tested is operating at or above the maximum sludge charging rate at which such facility will be operated and burning sewage sludge representative of normal operation, and/or under such other conditions as the Administrator shall specify in order to achieve valid representative test results.

(c) Compliance with the standard shall be determined by sampling and observing undiluted gases. If air or other gaseous diluent is added prior to a sampling or observation point, the owner or operator shall determine the amount of dilution by a means acceptable to the Administrator.

(d) The reference methods for conducting performance tests are appended to this part.

(1) Method 5 shall be used for determining concentration of particulate matter and moisture, method 1 for traversing, method 2 for determining the volumetric flow rate, and method 3 for gas analysis. The sampling time shall be not less than 60 minutes and not more than 150 minutes, and the minimum sampling rate shall be 0.9 dry standard cubic meter per hour (0.53 dscfm).

6. The appendix is amended by adding method 10 and method 11 as follows:

METHOD 10.—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability.*—1.1 *Principle.*—An integrated or grab gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a nondispersive infrared analyzer (NDIR) or equivalent.

1.2 *Applicability.*—This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a grab or an integrated sample is to be used.

2. *Range and sensitivity.*—2.1 *Range.*—0 to 1,000 ppm. 2.2 *Sensitivity.*—Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences.*—3.1 Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H_2O) and carbon dioxide (CO_2) are 3.5 percent H_2O per 7 ppm CO and 10 percent CO_2 per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H_2O per 25 ppm CO and 10 percent CO_2 per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. *Precision and accuracy.*—4.1 *Precision.*—The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 *Accuracy.*—The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. *Apparatus.*—5.1 Grab sample (fig. 10–1).

5.1.1 *Probe.*—Stainless steel or sheathed Pyrex[1] glass, equipped with a filter to remove particulate matter.

5.1.2 *Air-cooled condenser or equivalent.*—To remove any excess moisture.

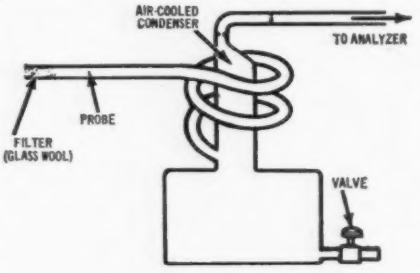

Figure 10-1. Grab-sampling train.

5.2 *Integrated sample (fig. 10–2).*—5.2.1 *Probe.*—Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 *Air cooled condenser or equivalent.*—To remove any excess moisture.

5.2.3. *Valve.*—Needle valve, or equivalent, to adjust flow rate.

5.2.4 *Pump.*—Leak-free, diaphragm type, or equivalent, to transport gas.

5.2.5 *Rate meter.*—Rota meter, or equivalent, to measure a flow range from 0 to 1.0 1pm (0.035 CFM).

5.2.6. *Flexible bag.*—Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft³). Leak test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

5.2.7 *Pilot tube.*—Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

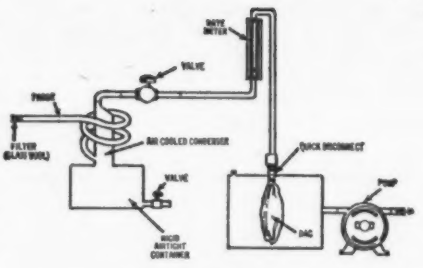

Figure 10-2. Integrated gas-sampling train.

5.3 Analysis (fig. 10–3).

5.3.1 *Carbon monoxide analyzer.*—Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturers specifications and those described in this method.

5.3.2 *Drying tube.*—To contain approximately 200 g of silica gel.

5.3.3 *Calibration gas.*—Refer to paragraph 6.1.

5.3.4 *Filter.*—As recommended by NDIR manufacturer.

5.3.5 *CO_2 removal tube.*—To contain approximately 500 g of ascarite.

5.3.6 *Ice water bath.*—For ascarite and silica gel tubes.

5.3.7 *Valve.*—Needle valve, or equivalent, to adjust flow rate.

5.3.8 *Rate meter.*—Rotameter or equivalent to measure gas flow rate of 0 to 1.0 1pm (0.035 CFM) through NDIR.

5.3.9 *Recorder (optional).*—To provide permanent record of NDIR readings.

5.3.10 *Orsat analyzer, or equivalent.*

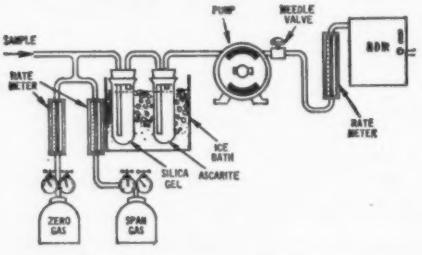

Figure 10-3. Analytical equipment.

6. *Reagents.*—6.1 *Calibration gases.*—Known concentration of CO nitrogen (N_2) for instrument span, prepurified grade of N_2 for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard.

6.2 *Silica gel.*—Indicating type, 6 to 16 mesh, dried at 177 (350° F) for 2 hours.

6.3 *Ascarite.*—Commercially available.

7. *Procedure.*—7.1 *Sampling.*—7.1.1 *Grab sampling.*—Set up the equipment as shown in figure 10–1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and draw sample into the analyzer. Allow 5 minutes for the system to stabilize and record the analyzer reading. (See §§ 7.2 and 8). Determine CO_2 content of the gas using the method 3 grab sample procedure (36 FR 24886).

7.1.2 *Integrated sampling.*—Evacuate the flexible bag. Set up the equipment as shown in figure 10–2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure

that all connections are leak free. Sample at a rate proportional to the stack velocity. Determine the CO_2 content of the gas in the bag using the method 3 integrated sample procedure (36 FR 24886).

7.2 *CO Analysis.*—Assemble the apparatus as shown in figure 10–3, calibrate the instrument, and perform other required operations as described in paragraph 8. Purge sample with N_2 prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on table 10–1.

TABLE 10–1.—*Field data*

Location _____ Comments:
Test _____
Date _____
Operator _____

Clock time:	*Rotameter setting, 1 pm (ft³ min)*

8. *Calibration.*—Assemble the apparatus according to figure 10–3. Generally an instrument requires a warmup period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of 1 hour for warmup. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and CO_2 removal tube, to insure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

9. *Calculations.*—9.1 *Concentration of carbon monoxide.*—Calculate the concentration of carbon monoxide in the stack using equation 10–1.

$$C_{CO_{stack}} = C_{CO_{NDIR}} (1 - F_{CO_2}) \quad \text{equation 10–1}$$

where:

$C_{CO_{stack}}$ = Concentration of CO in stack, ppm by volume (dry basis).
$C_{CO_{NDIR}}$ = Concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).
F_{CO_2} = Volume fraction of CO_2 in sample, i.e., percent CO_2 from Orsat analysis divided by 100.

10. *Bibliography.*—The Intertech NDIR–CO Analyzer by Frank McElroy. Presented at the 11th Methods Conference in Air Pollution, University of California, Berkeley, Calif., Apr. 1, 1970.

Jacobs, M. B., et al., JAPCA *9*, No. 2, 110–114, Aug. 1959.

MSA LIRA Infrared Gas and Liquid Analyzer Instruction Book, Mine Safety Appliances Co., Pittsburgh, Pa.

Beckman Instruction 1635B, Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instrument Co., Fullerton, Calif.

Continuous CO Monitoring System, Model A 5611, Intertech Corp., Princeton, N.J.

Bendix—UNOR Infrared Gas Analyzers. Ronceverte, W. Va.

ADDENDA

A. PERFORMANCE SPECIFICATIONS FOR NDIR CARBON MONOXIDE ANALYZERS

Range (minimum)	0–1,000 ppm.
Output (minimum)	0–10 mv.
Minimum detectable sensitivity.	20 ppm.
Rise time, 90 percent (maximum).	30 seconds.
Fall time, 90 percent (maximum).	Do.
Zero drift (maximum)	10% in 8 hours.
Span drift (maximum)	Do.
Precision (minimum)	±2% of full scale.
Noise (maximum)	±1% of full scale.
Linearity (maximum deviation).	2% of full scale.
Interference rejection ratio.	CO_2—1,000 to 1, H_2O—500 to 1.

B. DEFINITIONS OF PERFORMANCE SPECIFICATIONS

Range.—The minimum and maximum measurement limits.

Output.—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

Full scale.—The maximum measuring limit for a given range.

Minimum detectable sensitivity.—The smallest amount of input concentration that can be detected as the concentration approaches zero.

Accuracy.—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

Time to 90 percent response.—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

Rise time (90 percent).—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

Fall time (90 percent).—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

Zero drift.—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

Span drift.—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

Precision.—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

Noise.—Spontaneous deviations from a mean output not caused by input concentration changes.

Linearity.—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

METHOD 11.—DETERMINATION OF HYDROGEN SULFIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*—1.1 *Principle.*—Hydrogen sulfide (H_2S) is collected from the source in a series of midget impingers and reacted with alkaline cadmium hydroxide [$Cd(OH)_2$] to form cadmium sulfide (CdS). The precipitated CdS is then dissolved in hydrochloric acid and absorbed in a known volume of iodine solution. The iodine consumed is a measure of the H_2S content of the gas.

1.2 *Applicability.*—This method is applicable for the determination of hydrogen sulfide emissions from stationary sources only when specified by the test procedures for determining compliance with the new source performance standards.

2. *Apparatus.*—2.1 *Sampling train.*—2.1.1 *Sampling line.*—0.64 cm (one-fourth inch) Teflon[1] tubing to connect sampling train to sampling valve, with provisions for heating to prevent condensation. A pressure reducing valve prior to the Teflon sampling line may be required depending on sampling stream pressure.

2.1.2 *Impingers.*—Four midget impingers, each with 30-ml capacity, or equivalent.

2.1.3 *Ice bath container.*—To maintain absorbing solution at a constant temperature.

2.1.4 *Silica gel drying tube.*—To protect pump and dry gas meter.

2.1.5 *Needle valve, or equivalent.*—To adjust gas flow rate.

2.1.6 *Pump.*—Leak free, diaphragm type, or equivalent, to transport gas. (Not required if sampling stream under positive pressure.)

2.1.7 *Dry gas meter.*—Sufficiently accurate to measure sample volume to within 1 percent.

2.1.8 *Rate meter.*—Rotameter, or equivalent, to measure a flow rate of 0 to 2.83 lpm (0.1 ft³/min).

2.1.9 *Graduated cylinder.*—25 ml.

2.1.10 *Barometer.*—To measure atmospheric pressure within ±2.5 mm (0.1 in.) Hg.

2.2 *Sample recovery.*—2.2.1 *Sample container.*—500-ml glass stoppered iodine number flask.

2.2.2 *Pipette.*—50-ml volumetric type.

2.2.3 *Beakers.*—250 ml.

2.2.4 *Wash bottle.*—Glass.

2.3 *Analysis.*—2.3.1 *Flask.*—500-ml glass stoppered iodine number flask.

2.3.2 *Burette.*—One 50 ml.

2.3.3 *Flask.*—125-ml. conical.

3. *Reagents.*—3.1 *Sampling.*—3.1.1 *Absorbing solution.*—Cadmium hydroxide (Cd $(OH)_2$). Mix 4.3 g cadmium sulfate hydrate ($3\,CdSO_4.8H_2O$) and 0.3 g of sodium hydroxide (NaOH) in 1 liter of distilled water (H_2O). Mix well.

NOTE.—The cadmium hydroxide formed in this mixture will precipitate as a white suspension. Therefore, this solution must be thoroughly mixed before using to insure an even distribution of the cadmium hydroxide.

3.2 *Sample recovery.*—3.2.1 10 percent by weight hydrochloric acid solution (HCl)—Mix 230 ml of concentrated HCl (specific gravity 1.19) and 770 ml of distilled H_2O.

3.2.2 *Iodine Solution, 0.1 N.*—Dissolve 24 g potassium iodide (KI) in 30 ml of distilled H_2O in a 1-liter graduated cylinder. Weigh 12.7 g of resublimed iodine (I_2) into a weighing bottle and add to the potassium iodide solution. Shake the mixture until the iodine is completely dissolved. Slowly dilute the solution to 1 liter with distilled H_2O, with swirling. Filter the solution, if cloudy, and store in a brown glass-stoppered bottle.

3.2.3 *Standard Iodine Solution, 0.01 N.*—Dilute 100±0.01 ml of the 0.1 N iodine solution in a volumetric flask to 1 liter with distilled water.

Standardize daily as follows: Pipette 25 ml of the 0.01 N iodine solution into a 125-ml conical flask. Titrate with standard 0.01 N thiosulfate solution (see paragraph 3.3.2) until the solution is a light yellow. Add a few drops of the starch solution and continue titrating until the blue color just disappears.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

From the results of this titration calculate the exact normality of the iodine solution (see paragraph 5.1).

3.2.4 *Distilled, deionized water.*—3.3 *Analysis.*—3.3.1 *Sodium thiosulfate solution, standard 0.1 N.*—For each liter of solution, dissolve 24.8 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in distilled water and add 0.01 g of anhydrous sodium carbonate (Na_2CO_3) and 0.4 ml of chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes, and store in a glass-stoppered glass bottle.

Standardize frequently as follows: Weigh into a 500-ml volumetric flask about 2 g of potassium dichromate ($K_2Cr_2O_7$) weighed to the nearest milligram and dilute to the 500-ml mark with distilled H_2O. Use dichromate which has been crystallized from distilled water and oven-dried at 182° to 199° C (360° to 390° F.). Dissolve approximately 3 g of potassium iodide (KI) in 50 ml of distilled water in a glass-stoppered, 500-ml conical flask, then add 5 ml of 20-percent hydrochloric acid solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of distilled water, washing down the sides of the flask with part of the water. Swirl the solution slowly and titrate with the thiosulfate solution until the solution is light yellow. Add 4 ml of starch solution and continue with a slow titration with the thiosulfate until the bright blue color has disappeared and only the pale green color of the chromic ion remains. From this titration, calculate the exact normality of the sodium thiosulfate solution (see paragraph 5.2).

3.3.2 *Sodium thiosulfate solution, standard 0.01 N.*—Dilute 100 ±0.01 ml of the standard 0.1 N thiosulfate solution in a volumetric flask to 1 l with distilled water.

3.3.3 *Starch Indicator Solution.*—Suspend 10 g of soluble starch in 100 ml of distilled water and add 15 g of potassium hydroxide pellets. Stir until dissolved, dilute with 900 ml of distilled water, and let stand 1 hour. Neutralize the alkali with concentrated hydrochloric acid, using an indicator paper similar to Alkacid test ribbon, then add 2 ml of glacial acetic acid as a preservative.

Test for decomposition by titrating 4 ml of starch solution in 200 ml of distilled water with the 0.01 N iodine solution. If more than 4 drops of the 0.01 N iodine solution are required to obtain the blue color, make up a fresh starch solution.

4. *Procedure.*—4.1 Sampling.—4.1.1 Assemble the sampling train as shown in figure 11-1, connecting the 4 midget impingers in series. Place 15 ml of the absorbing solution in each of the first three impingers, leaving the fourth dry. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 21° C (70° F) or less.

4.1.2 Purge the connecting line between the sampling valve and the first impinger. Connect the sample line to the train. Record the initial reading on the dry gas meter as shown in table 11-1.

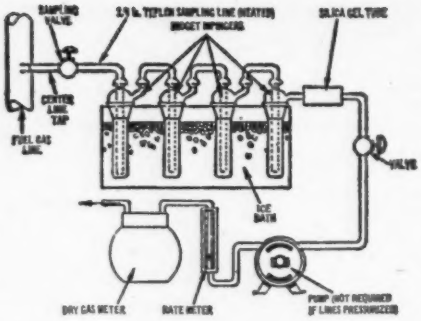

Figure 11-1. H_2S sampling train.

TABLE 11-1.—*Field data*

Location..... Comments:
Test.....
Date.....
Operator.....
Barometric pressure.....

Clock time	Gas volume through meter (V_m), liters (ft^3)	Rotameter setting, 1 pm (ft^3/min)	Meter temperature, °C (°F)

4.1.3 Open the flow control valve and adjust the sampling rate to 1.13 lpm (0.04 cfm). Read the meter temperature and record on table 11-1.

4.1.4 Continue sampling for 10 minutes or until the yellow color of cadmium sulfide is visible in the third impinger. At the end of this time, close the flow control valve and read the final meter volume and temperature.

4.1.5 Disconnect the impinger train from the sampling line and cap the open ends. Remove to the sample clean-up area.

4.2 Sample Recovery.

4.2.1 Pipette 50 ml of 0.01 N iodine solution into a 250-ml beaker. Add 50 ml of 10 percent HCl to the solution. Mix well.

4.2.2 Carefully transfer the contents of all impingers to a 500-ml iodine number flask. Stopper the flask.

4.2.3 Rinse all impingers and connecting glassware with three portions of the acidified iodine solution. Use the entire 100 ml of acidified iodine for this purpose. Immediately after pouring the acidified iodine into an impinger, stopper it and shake for a few moments before transferring the rinse to the iodine number flask. Do not transfer any rinse portions from one impinger to another; transfer it directly to the iodine number flask. Once acidified iodine solution has been poured into any glassware containing cadmium sulfide sample the container must be tightly stoppered at all times except when adding more solution, and this must be done as quickly and carefully as possible. After adding any acidified iodine solution to the iodine number flask allow a few minutes for absorption of the H_2S into the iodine before adding any further rinses.

4.2.4 Follow this rinse with two more rinses using distilled water. Add the distilled water rinses to the iodine number flask. Stopper the flask and shake well. Allow about 30 minutes for absorption of the H_2S into the iodine, then complete the analysis titration.

CAUTION.—Keep the iodine number flask stoppered except when adding sample or titrant.

4.2.5 Prepare a blank in an iodine number flash using 45 ml of the absorbing solution, 50 ml of 0.01 N iodine solution, and 50 ml of 10 percent HCl. Stopper the flask, shake well and analyze with the samples.

4.3 Analysis.

NOTE.—This analysis titration should be conducted at the sampling location in order to prevent loss of iodine from the sample.

4.3.1 Titrate the solution in the flask with 0.01 N sodium thiosulfate solution until the solution is light yellow. Add 4 ml of the starch indicator solution and continue titrating until the blue color just disappears.

4.3.2 Titrate the blanks in the same manner as the samples.

5. *Calculation.*—5.1 *Normality of the standard iodine solution.*

$$N_T = \frac{N_T V_T}{V_I} \quad \text{equation 11-1}$$

where:

N_I = Normality of iodine, g-eq/liter.
V_I = Volume of iodine used, ml.
N_T = Normality of sodium thiosulfate, g-eq/liter.
V_T = Volume of sodium thiosulfate used, ml.

5.2 *Normality of the standard thiosulfate solution.*

$$N_T = 1.02 \frac{W}{V_T} \quad \text{equation 11-2}$$

where:

W = Weight of $K_2Cr_2O_7$ used, g.
V_T = Volume of $Na_2S_2O_3$ used, ml.
N_T = Normality of standard thiosulfate solution, g-eq/liter.

$$1.02 = \text{conversion factor} = \frac{(3 \text{ eq } I_2/\text{mole } K_2CrO_7)\ (1{,}000 \text{ ml/l})}{(294.2 \text{ g } K_2Cr_2O_7/\text{mol})\ (10\text{e aliquot factor})}.$$

5.3 *Dry gas volume.*—Correct the sample volume measured by the dry gas meter to standard conditions (21°C (70°F) and 760 mm (29.92 inches) Hg) by using equation 11-3.

$$V_{m_{std}} = V_m \left(\frac{I_{std}}{T_m}\right)\left(\frac{P_{Bar}}{P_{std}}\right) \quad \text{equation 11-3}$$

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), 1 (scf).
V_m = Volume of gas sample through the dry gas meter (meter conditions), 1 (cu. ft.).
T_{std} = Absolute temperature at standard conditions, 294° K (530° R).
T_m = Average dry gas meter temperature, °K (°R).
P_{Bar} = Barometric pressure at the orifice meter, mm Hg.
P_{std} = Absolute pressure at standard conditions, 760 mm Hg.

5.4 Concentration of H_2S. Calculate the concentration of H_2S in the gas stream at standard conditions using equation 11-4.

$$C_{H_2S} = \frac{K[(V_I N_I)_{sample} - (V_I N_I - V_T N_T)_{blank}]}{V_{m_{std}}} \quad \text{equation 11-4}$$

where (metric units):

C_{H_2S} = Concentration of H_2S at standard conditions (mg/Nm^3)

K = Conversion factor = $17.0 \times 10^3 = \frac{(34.07 \text{ g/mole } H_2S)\ (1{,}000\ 1/m^3)\ (1000 \text{ mg/g})}{(1{,}000 \text{ ml/l})\ (2\ H_2S \text{ eq/mole})}$

V_I = Volume of standard iodine solution, ml.

N_I = Normality of standard iodine solution, g-eg/liter.

V_T = Volume of standard sodium thiosulfate solution, ml.

N_T = Normality of standard sodium thiosulfate solution, g-eq/liter.

$V_{m_{std}}$ = Dry gas volume at standard conditions, liters.

Or

where (English units):

$K = 0.263 = \frac{17.0\ (15.43 \text{ gr/g})}{(1{,}000\ 1/m^3)}$

$V_{m_{std}}$ = scf

C_{H_2S} = gr/dscf

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6. *References.*—American Petroleum Institute, Determination of Hydrogen Sulfide, Ammoniacal Cadmium Chloride Method, API Method 772–54.

National Gas Processors Association, NGPA Publication 2265–65, Tentative Method for Determination of Hydrogen Sulfide and Mercaptan Sulfur in Natural Gas.

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